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Characterisation of Residual Stress in Dielectric Films Studied by Automated Wafer Mapping

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***Abstract* - SU-8 is a negative epoxy based photoresist, which is widely used as a structural and dielectric layer in the fabrication of MEMS devices. However this material normally develops high levels of stress during processing. This paper reports detailed quantitative data following previous work, where Parylene-C has been proposed as a possible replacement for SU-8. In particular, this paper details the characterisation of residual stress in (i) SU-8 films as a function of processing temperatures and (ii) post-processing thermal treatment of Parylene-C. This characterisation includes wafer mapping strain using rotating pointer arm test structures, and deriving the stress from independent measurements of strain and Young's modulus.**

I. Introduction

SU-8 has a wide range of applications as a structural and dielectric layer in MEMS devices [1]. However, intrinsic problems with mechanical stress resulting from shrinkage during the cross linking process [1-3], can often affect the reliability and the total yield of the fabricated devices. For this reason, it is important to be able to characterise the stress in this material, at various processing conditions, as well as identifying any possible candidate materials to replace SU-8.

One possible replacement is Parylene-C, which is a polymer material that is widely used as a protective coating on printed circuit boards (PCB) [4]. This material can be conformally deposited from vapour phase at room temperature, and acts as a dielectric with outstanding barrier and mechanical properties, as a result of inherent low stress deposition [5]. In previous work [6], it has been demonstrated that Parylene can be successfully used instead of SU-8 as the inter-coil dielectric in an integrated micro-inductor process.

This paper reports on the characterisation of the residual stress in SU-8 and Parylene-C films as a result of process parameter variation. The measurements include the spatial variation of strain, mapped over the surface of 3 inch wafers. This involves measuring an array of 720 micro-fabricated rotating pointer arm test structures. Stress is derived from independent measurements of strain and Young's modulus [7].

In addition, test structure have been designed to provide quantitative information from a peel test to characterise the adhesion force of Parylene and SU-8, to a number of materials commonly used in MEMS processing.

II. Test Structure Design

The mechanical test structures used in this study consist of a rotating pointer arm structure, and two lateral expansion arms. The expansion arms are offset at the point where they connect to the pointer arm. Thus, expansion or contraction in these arms results in rotation of the pointer arm in the plane of the layer. Figure 1 shows a schematic of the test structure, which has been reported previously [1]. In this design clockwise rotation indicates a tensile stress, whilst counter clockwise rotation indicates a compressive stress.

Maximum rotation has been found previously to occur when the ratio between expansion arm's offset and arm's width is 1.5 [1]. However, when characterising strain in films with low Young's modulus (<10 GPa) it is advantageous to increase this ratio to 2 in order to reduce the maximum degree of rotation [7].

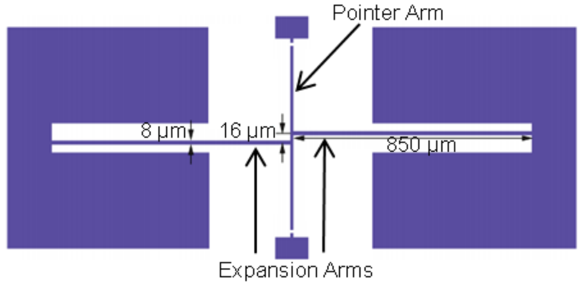


Figure 1: Schematic of strain test structure.

III. Fabrication

The SU-8 rotating strain test structures used in this investigation were patterned on top of silicon wafers coated with 1µm of thermally grown silicon dioxide, covered by 700 nm of LPCVD deposited polysilicon. Two types of SU-8 (SU-8 5 and SU-8 3005), were characterised to investigate the effect of the hard bake (HB) temperature and cool down rate on their residual stress. Two hard bake temperatures 200˚C (standard) and 150˚C were evaluated using a cool down rate at the end of the process of 2.83˚C/min.

The Parylene strain structures were fabricated on the same silicon/SiO_2/polysilicon substrate. A 5 µm thick Parylene film was deposited, and test structures were then patterned using a RIE based oxygen plasma process, with a titanium hard mask. The resulting structures were then annealed at temperatures of 70˚C, 140˚C, and 200˚C to investigate the effect of thermal budgets on the film's residual stress.

In all cases the polysilicon sacrificial layer used to release the structures was removed using XeF_2 vapour. Figure 2 shows a photograph of a released Parylene strain indicator structure.

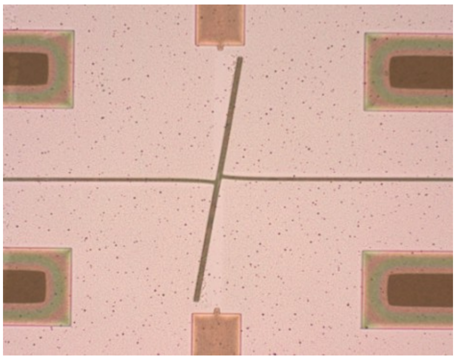


Figure 2: A released Parylene strain indicator structure (note: the expansion arms are anchored out of view).

IV: Nano-Indentation Measurements

Nano-indentation measurements were used to determine Young's modulus. The nanoindenter used was a Hysitron triboindenter with a Berkovich diamond tip, which indents into the surface of the material using a controlled force regime while measuring the response of the film. The load applied by the tip is ramped to 2 mN at a rate of 0.4 mN/sec, then held for 10 seconds, and ramped down at a rate of 0.4 mN/sec. Typical measured indentation curves for SU-8 5 and Parylene are presented in Figure 3. The data from the unloading segment of this curve is used to extract the Young's modulus using the curve fitting procedure detailed in [8].

Each three inch wafer was diced into 1 cm^2 chips, and 4 indentation measurements were taken on each of these chips. The average Young's modulus values measured for all SU-8 and Parylene samples are presented in figure 4.

The measured Young's modulus values for SU-8 ranged from 4.17 GPa (SU-8 3005 hard-baked at 200˚C with gradual cool down phase) to 5.49 GPa (SU-8 5 hard-baked at 200˚C). In the literature, these values typically range between 3.5 and 7.0 GPa [9], which covers the values determined in this exercise.

Values of Young's modulus for Parylene reported in the literature range between 2.00GPa and 5.29GPa [10, 11]. This compares with the parameters measured in this work, which were 1.01 GPa to 2.87 GPa for non-annealed and annealed samples at 140˚C respectively.

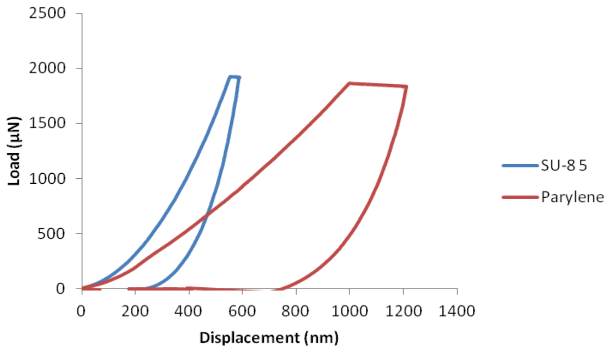
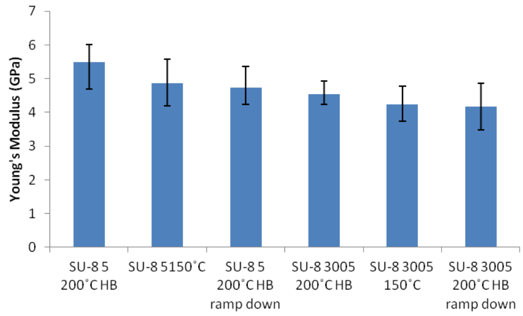
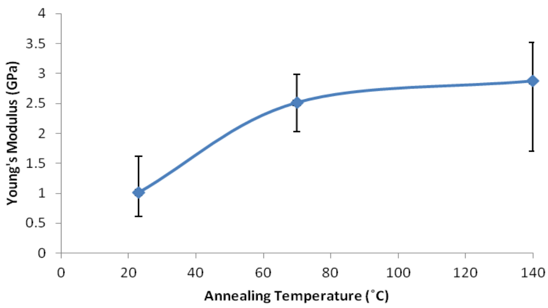


Figure 3: Indentation curves for SU-8 5 hard baked at 200˚C and non-annealed Parylene .



(a)



(b)

Figure 4: Young's modulus values with respect to annealing temperature: (a) SU-8, (b) Parylene, error bars indicate range.

V. Finite Element Modelling

Finite element (FE) modelling has been used to combine strain data with Young's modulus measurements, to compute values for residual stress. This FE model simulates the response of the test structure for a given stress and Young's modulus. The strain data obtained from the simulated curves (figure 5) was then correlated with strain data obtained from strain test structure measurements, in order to determine stress in the film.

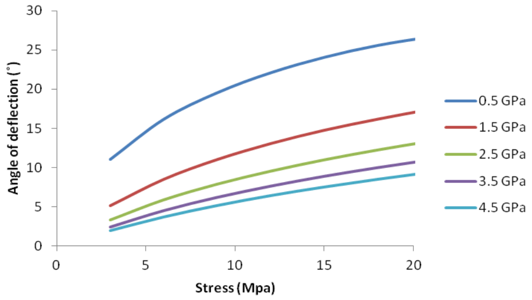


Figure 5: Simulated results of residual stress mapped against angle of rotation for a range of different Young's modulus values.

VI. Stress Wafer Mapping

By correlating strain measurements from the pointer arm test structures with the simulated response of the strain indicator structure (figure 5), it has been possible to determine the stress in the Parylene and SU-8 films. Figure 6 presents the average stress for each of the SU-8 and Parylene films.

In addition, by correlating the position of strain and nano-indentation measurements on the wafer it has been possible to determine local values of stress, and subsequently wafer map strain; Young's modulus; and stress for each film. These wafer maps are presented in figure 7.

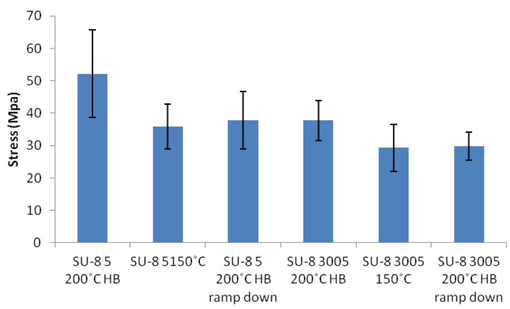
It is clear from figures 6 and 7 that the highest value of stress was developed in SU-8 5 hard baked at 200˚C (σ = 52.19 MPa). It is also interesting to note that SU-8 films that were hard baked at 150˚C exhibited approximately the same average stress values (~36 MPa for SU-8 5 and 29 MPa for SU-8 3005) as SU-8 films which were hard baked at 200˚C followed by a gradual ramp down. These results indicate that gradually cooling further to hard bake results in a 27% and 21% reduction in stress for SU-8 5 and SU-8 3005 respectively. SU-8 3005 also exhibited a 27.7%, 18.4%, 21.5% lower average stress compared to SU-8 5 samples which had undergone the same processing conditions shown in figure 6(a).

Wafer maps of SU-8 also present a large variation in stress. The worst case is with SU-8 5 for a 200˚C hard bake, where the maximum stress variation is 74MPa.

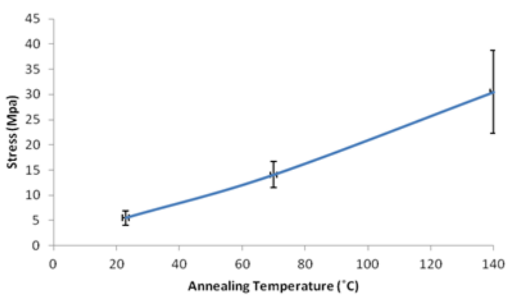
Non-annealed Parylene samples initially exhibit a lower stress level than SU-8 samples (5.52 MPa), which increases to 14.07 and 30.05 MPa when annealed at 70˚C and 140˚C respectively. However, it is important to note that Parylene annealed at 140°C develops a comparable level of stress with SU-8 3005 annealed at 150˚C or 200˚C followed by gradual cooling.

When Parylene is annealed at 200˚C all the stress test structures fractured during the release processing step, and it was therefore not possible to obtain any quantitative information. It is postulated that their rotation angle would be significantly larger than of those annealed at 140˚C and as a result they experienced a high degree of stress resulting in fracture. The Young's modulus of Parylene annealed at 200˚C was found to be 5.2 GPa. Therefore, a significant increase of 414% in Young's modulus between non-annealed Parylene and Parylene annealed at 200˚C suggests a significantly higher stress resulting from exposure to higher temperatures. This stress is assumed to exceed the yield strength value for Parylene (59 MPa) [12]. Therefore when considering integrating Parylene into device architecture, thermal treatment must be carefully considered.

Wafer maps of non-annealed and annealed at 70˚C Parylene films appear to exhibit uniform mechanical properties. In this case the maximum variation in stress across the wafers is 10 and 15 MPa for non-annealed and 70˚C annealed samples. Wafers which have been annealed at 140˚C show a 440% and 260% higher variation of 54 MPa, when compared to non-annealed and 70˚C annealed Parylene films. This level of variation is comparable to the 55 and 51 MPa values reported in SU-8 5 films which have been hard baked at 150˚C and 200˚C with gradual cool down. It has been observed that the stress in the Parylene film annealed at 140˚C has developed a stress gradient across the wafer, from low stress in the bottom left of the map (approximately 20 MPa) to greater levels of stress in the centre and top right of the map (approximately 40 MPa). As other measurements indicate that higher temperatures result in greater levels of residual stress it is thought that this effect could be the result of a significant temperature gradient across the wafer, when annealed on the hotplate.



(a)



(b)

Figure 6: Average stress data for (a) SU-8 and (b) Parylene, error bars indicate standard deviation.

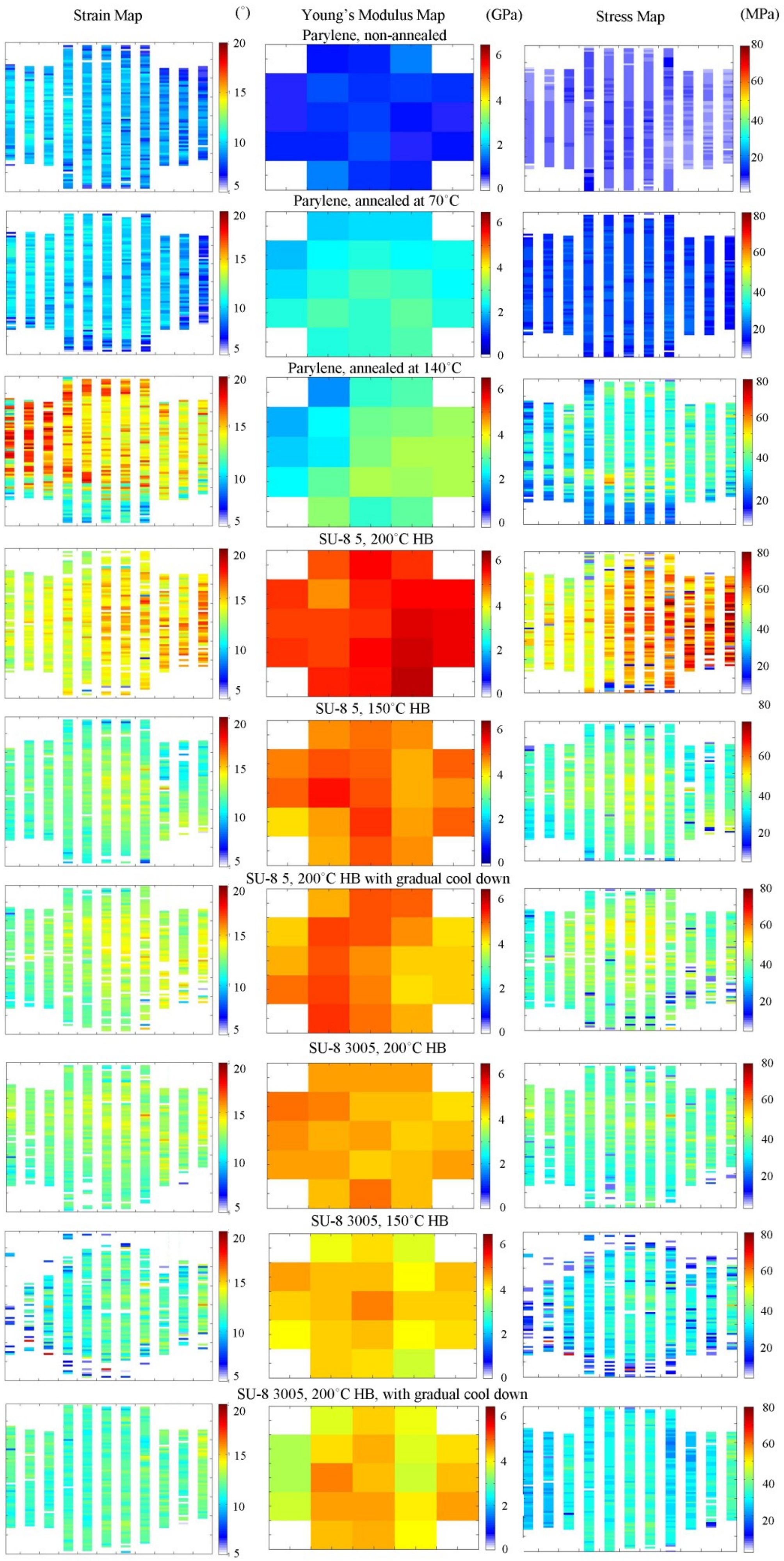


Figure 7: Wafer maps of strain, Young's modulus and stress for all SU-8 and Parylene films.

VII. Adhesion Testing

In addition to stress, the adhesion strength of SU-8 and Parylene deposited on materials commonly used in the fabrication of MEMS (e.g. silicon dioxide, copper, titanium and NiFe) has been considered. In this study, test structures consisting of long strips (5 cm) of SU-8/Parylene were fabricated. The design consisted of a 1.5 cm long section of this strip being mechanically anchored to the material under test (MUT), while a 3.5 cm of the strips length was released by removing the polysilicon sacrificial layer using a XeF_2 vapour etchant. Figure 8 (a) presents a photograph of the fabricated strips with 8(b) showing cross-section of the fabricated structure.

After fabrication, a tensile testing setup was used to carry out a peel test by fixing the wafer to a chuck and, using the expansion arm of the tensile testing rig to pull the released section of the strip at 90˚ to the substrate. A diagram of this setup is presented in figure 9.

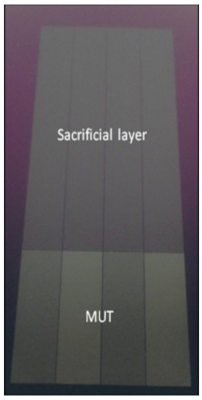
Using this method it was possible to obtain one measurement of Parylene, figure 10, where the material under test was silicon dioxide, indicating that this technique can be used to measure the adhesion force of thin films. However, when testing all other samples, including others with silicon dioxide, the Parylene broke when the pull force exceeded approximately 2.5 N. Thus, the conclusion can be drawn that the adhesion force for Parylene, for all base materials tested is greater than the yield strength of Parylene. However, it is suspected that bending the strip through 90˚ may have damaged the Parylene significantly reducing its yield strength. It should also be noted that the array of 10 µm etch holes every 200 µm for the sacrificial polysilicon material removal may also have contributed to the lower yield strength.

It proved not to be possible to use this technique to test SU-8 samples as the released strip was too brittle to be bent through 90^o and broke during the clamping process. Therefore no useful numerical data has been recorded for SU-8 samples.

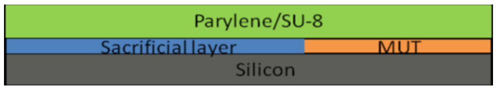
VIII. Conclusion and Future Work

In this paper the strain, Young's modulus and stress have been characterised, for Parylene and SU-8 films with different thermal histories, by employing the use of strain indictor test structures and nano-indentation. By correlating the position of strain and Young's modulus measurements on the wafer it has been possible to wafer map strain, Young's modulus and stress for each film.

It has been established that by annealing Parylene the average stress in these films increase by 116.4% and 452% for films annealed at 70˚C and 140˚C respectively. In addition, all Parylene strain indicator structures which were annealed at 200˚C fractured, indicating that the stress in the film is greater than the yield strength. Therefore, when considering the use of Parylene, thermal treatment must be carefully considered.



(a)



(b)

Figure 8: (a) fabricated adhesion test structures (before release), (b) cross-section of fabricated test structures (before release).

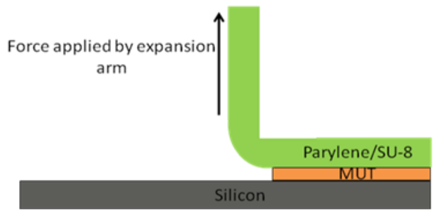


Figure 9: Test structure setup for tensile testing.

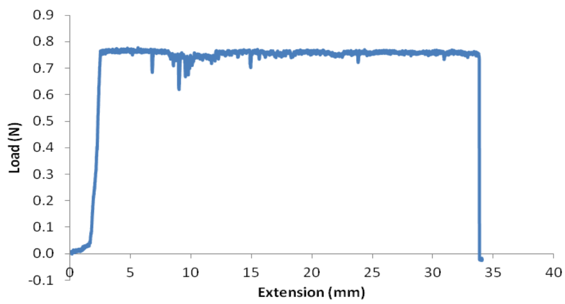


Figure 10: Peel test result for Parylene on silicon dioxide.

The strain, Young's modulus and stress have been characterised for two types of SU-8 (SU-8 5 and SU-8 3005) and the effect of hard bake temperature and gradual cooling on residual stress has been investigated. It is interesting to note that a gradual cool down, further to a 200˚C hard bake, reduced the average residual stress by 27% and 21% for SU-8 5 and SU-8 3005 respectively.

Reducing the hard bake temperature to 150˚C also reduced the average residual stress by 31% and 17%, for SU-8 5 and SU-8 3005 respectively. It is clear that these results are comparable to SU-8 samples which were hard baked at 200˚C with a gradual cool down. For samples which were annealed at 150˚C and 200˚C with a gradual cool down, it is thought that the reduced stress is the result of a lower degree of shrinkage following the hard bake stage.

SU-8 3005 samples consistently exhibited a lower stress than SU-8 5 samples (32% for SU-8 5 and SU-8 3005 samples, with 200˚C hard bake, without gradual cool down).

Adhesion tests have been carried out using a tensile testing rig, and it has been found that the adhesion force is greater than the yield strength. Parylene when pulled at 90^{o} to the substrate orientation.

Future work will involve the characterisation of SU-8 and Parylene films under different humidity conditions.

IX. ACKNOWLEDGEMENTS

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X. REFERENCES

[1] S. Smith, N. L. Brockie, J. Murray, G. Schiavone, C. J. Wilson, A. B. Horsfall, J. G. Terry, J. T. M. Stevenson, A. R. Mount, A. J. Walton, "Fabrication and Measurement of Test Structures to Monitor Stress in SU-8 Films," *IEEE Trans. Semicond. Manuf.,* vol. 25, no. 3, pp. 346–354, 2012

[2] J. Hammacher, A. Fuelle, J. Flaemig, J. Saupe, B. Loechel, J.. Grimm, "Stress engineering and mechanical properties of SU-8-layers for mechanical applications," *Microsystem Technologies*, Vol 14, No 9., pp. 1515-1523, 2008

[3] R. Feng, R. J. Farris "Influence of processing conditions on the thermal and mechanical properties of SU8 negative photoresist coatings," J. Micromech. Microeng., vol. 13, pp. 80-88, 2003.

[4] *M.E.* Cosens. "Parylene conformal coatings [PCB protection]" *Electronic Production*, vol. 12, no. 3, pp. 12-13, 1983.

[5] S. Dabral, J. Van Etten, X. Zhang, C. Apblett, G. -R. Yang, P. Ficalora, J. F. McDonald, "Stress in thermally annealed parylene films," *J. Electronic Material.,* vol. 21, no. 10, pp 989-994, 1992.

[6] R. Walker, E. Sirotkin, I. Schmueser, J. G. Terry, S. Smith, J.T.M. Stevenson, A.J. Walton, "Characterisation and integration of Parylene as an insulating structural layer for high aspect ratio electroplated copper coils," *ICMTS,* pp. 7-12, 2013.

[7] G. Schiavone, M.P.Y. Desmulliez, S. Smith, J. Murray, E. Sirotkin, J.G. Terry, A.R. Mount, A.J. Walton, "Quantitative wafer mapping of residual stress in electroplated NiFe films using independent strain and Young's modulus measurements," *ICMTS*, pp.105-110, 2012

[8] W.E. Oliver, G.M. Pharr, "Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology", *J. Mater. Res*, 2004

[9] A.T. Al-Halhouli, I. Kampen, T. Krah, S. Büttgenbach, "Nanoindentation testing of SU-8 photoresist mechanical properties", Microelectronic Engineering, Vol. 85, No 5–6, pp 942-944, 2008

[10] J.G. Cham, J. G. Cham, Z. Nenadic, S. Musallam, Y. C. Tai, J. W. Burdick, R. A. Andersen, "A New Multi-Site Probe Array with Monolithically Integrated Parylene Flexiable Cable for Neural Prosthesis," *Engineering in Medicine and Biology Society*, pp. 7114- 7117, 2005.

[11] D. Wright, D. Wright, B. Rajalingam, S. Selvarasah, Y. Ling, J. Yeh, R. Langer, Mehmet R. Dokmeci, A. Khademhosseini, "Reusable, Reversable, Sealable Parylene Membranes for Cell and Protein Patterning," *J. Biomedical Materials Research*, vol. 85A, no. 2, pp. 530- 538, 2007.

[12] V. C. Y. Shih, T.A. Harder, Y. C Tai, "Yield Strength of Thin-Film Parylene-C," *Symposium on Design, Test, Integration and Packaging of MEMS/MOEMS,* pp. 394-398, 2003.